



# Synthesis and Characterization of Binuclear Complexes of Metals Transition of N<sup>1</sup>,N<sup>4</sup>-Bis(1-(Pyridin-2- yl)Ethylidene)Succinohydrazide

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## Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

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## ABSTRACT

Six new dinuclear metal transition (Mn(II) (1), Fe(II) (2), Co(II) (3), Ni(II) (4), Cu(II) (5) and Zn(II) (6)) complexes have been synthesized from a new hexadentate Schiff base N<sup>1</sup>,N<sup>4</sup>-bis(1-(pyridin-2-yl)ethylidene)succinohydrazide (H<sub>2</sub>L). The ligand was characterized by elemental analysis, FTIR, UV-visible, <sup>1</sup>H NMR and <sup>13</sup>C NMR. The synthesized compounds have been investigated by

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elemental analysis, FTIR and UV-visible spectroscopies, molar conductance and room temperature magnetic moments measurement. The results show that the complexes 1-6 are dinuclear neutral electrolytes in DMF. Each of the two mono deprotonated tridentate arms of the ligand are coordinated to one metal ion through one iminolate oxygen atom, one azomethine nitrogen atom and one pyridine nitrogen atom. Octahedral geometries are proposed for all the complexes 1-6 formulated as  $[(ML)_2] \cdot nH_2O$ . The structure of the Ni(II) (4) complex is confirmed by X-ray crystallography study.

**Keywords:** FTIR; UV-visible; acetylpyridine; succinohydrazide; octahedral; X-ray.

## 1. INTRODUCTION

The interaction of transition metal ions with functionalized ligands is one of the most attractive and potentially useful areas of coordination chemistry [1–4]. Coordination chemistry of transition metals has attracted great interest in various fields of science and technology [1,5–7]. Ligands containing a hydrazone unit have been particularly studied in recent years, due to their interesting intrinsic properties and their significant chelating capacity [8–10]. Thus, these types of ligands are used to generate coordination compounds with diverse applications [11–14]. These Schiff bases can chelate different metal ions forming mononuclear [15,16], polynuclear [17,18] or heteropolynuclear [19–21] complexes. Dihydrazone ligands that are characterized by the presence of two hydrazonic groups linked by a spacer constitute good polydentate chelating agents that can present in several conformations to encapsulate metal ions, generating compounds with particular properties [22–24]. Thus, compounds with biological applications have been reported in the literature [25]. Coordination compounds with catalytic [26], magnetic [26], antimicrobial [27], antifungal [28], antituberculosis [29], and anticancer [30] properties are widely studied. Metal complexes of dihydrazones are also used in biomimetic chemistry for the study of various enzymes [31–33]. Our team has already synthesized and studied the structure of several compounds derived from hydrazonic ligands [34–38]. Therefore, in the continuity of our research work on transition metal complexes, we have synthesized a new dihydrazone ligand whose two arms are connected by a flexible spacer of type  $-(CH_2)_4-$ , which allows it to have a *syn* or *anti* conformation and a *cis* or *trans* configuration. This ligand allowed the synthesis of six new complexes whose structures are determined by a spectroscopic study (FTIR and UV-visible) and conductimetric and magnetic moments measurements at room temperature of

the complexes. The structure of the Ni(II) complex is confirmed by crystallography.

## 2. MATERIALS AND METHODS

### 2.1 Starting materials and Instrumentations

2-acetylpyridine, succinohydrazide, manganese chloride hexahydrate, iron chloride tetrahydrate, cobalt chloride hexahydrate, nickel chloride hexahydrate, copper chloride dihydrate and zinc chloride dihydrate were commercial products (from Aldrich) and were used without further purifications. The solvents were reagent grade and were purified by usual methods. Elemental analyses were carried out using a VxRio EL Instrument. The FTIR spectra were recorded on a FTIR Spectrum Two of Perkin Elmer ( $4000-400\text{ cm}^{-1}$ ). The UV-Visible spectra were run on a Perkin-Elmer UV/Visible spectrophotometer Lambda 365 (1000–200 nm). The  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra of the Schiff base were recorded in  $\text{DMSO}-d_6$  on a BRUKER 500 MHz spectrometer at room temperature using TMS as an internal reference. The molar conductance of  $10^{-3}\text{ M}$  solutions of the metal complexes in DMF were measured at  $25\text{ }^\circ\text{C}$  using a WTW LF-330 conductivity meter with a WTW conductivity cell. Magnetic measurements for complexes were performed at room temperature by using a Johnson Matthey scientific magnetic susceptibility balance (Calibrant:  $\text{Hg}[\text{Co}(\text{SCN})_4]$ ).

### 2.2 Synthesis of the Ligand $\text{H}_2\text{L}$

In a 250 mL round-bottomed flask containing 30 mL of methanol, 2 g (13.7 mmol) of succinohydrazide were added and the mixture was stirred for 30 min before adding 3.31 g (27.4 mmol) of 2-acetylpyridine. The mixture was refluxed for three hours. After cooling, the white solid was recovered by filtration and washed with ether (2 x 20 mL), then dried in the open air. M.P.  $252-254\text{ }^\circ\text{C}$ . Yield: 89%.  $^1\text{H}$  NMR ( $\text{DMSO}-d_6$ ,

$\delta$ (ppm) : 2.32 (s, 6H, -CH<sub>3</sub>), 3.05 (s, 4H, -CH<sub>2</sub>), 7.39 (m, 2H, H<sub>Ar</sub>), 7.79-7.85 (m, 2H, H<sub>Py</sub>), 8.02-8.06 (m, 2H, H<sub>py</sub>), 8.58 (s, 1H, O=C-NH), 10.70 (s, 1H, H-OC=N-). <sup>13</sup>C NMR (DMSO-d<sub>6</sub>,  $\delta$  (ppm)): 175.02 (-C=O), 155.64 (C=N), 149.02 (C<sub>Ar</sub>), 148.037 (-C=N<sub>py</sub>), 137.063(C<sub>Ar</sub>), 124.24(C<sub>Ar</sub>), 120.23(C<sub>Ar</sub>), 27.57(CH<sub>2</sub>), 12.25 (-CH<sub>3</sub>). <sup>13</sup>C NMR{DEPT135} (DMSO-d<sub>6</sub>,  $\delta$ (ppm)): 149.03, 137.06 (C<sub>Ar</sub>), 124.24 (C<sub>Ar</sub>), 120.23 (C<sub>Ar</sub>), 27.71 (CH<sub>2</sub>), 12.25 (-CH<sub>3</sub>).

### 2.3 Synthesis of the Complexes of H<sub>2</sub>L

0.106 g (0.3 mmol) of the ligand were introduced into a flask containing 25 mL of ethanol and 0.0252 g (0.6 mmol) of LiOH·H<sub>2</sub>O. An ethanol solution containing 0.6 mmol of transition metal chloride salt (M = Mn, Fe, Co, Ni, Cu, Zn) was added to the mixture. The mixture was refluxed for three hours. After cooling, the solid is recovered by filtration and then washed with cold ethanol (Table 1).

### 2.4 X-ray Structure Determination of Complex 4

Methanol solution of **4** was left to slow evaporation and yellow crystals suitable for X-ray analyze were formed after three weeks. The details of the X-ray crystal structure solution and refinement are given in Table 3. Measurements were made on a Bruker SMART CCD Area Detector. All data were corrected for Lorentz and polarization effects. Empirical absorption correction was applied. Complex scattering factors were taken from the program package *SHELXTL* [39]. The structures were solved by direct methods, which revealed the position of all non-hydrogen atoms. All the structures were refined on *F*<sup>2</sup> by a full-matrix least-squares procedure using anisotropic displacement parameters for all nonhydrogen atoms [40]. All hydrogen atoms were located in their calculated positions and refined using a riding model. The contribution of some disordered solvent to the scattering was removed using the *SQUEEZE* routine [41] in *PLATON*. The solvent contribution was not included in the reported molecular weight and density. Molecular graphics were generated using *ORTEP-3* [42].

## 3. RESULTS AND DISCUSSION

### 3.1 General Study

The infrared spectrum of the ligand shows in the high frequency region, medium intensity bands

between 3183 cm<sup>-1</sup> and 3079 cm<sup>-1</sup> which are assigned to the  $\nu_{N-H}$  stretching vibration of the amide moieties. The band at 3010 cm<sup>-1</sup> is assigned to the aromatic  $\nu_{C-H}$  vibration. The medium intensity bands between 1570 and 1410 cm<sup>-1</sup> are assigned to the  $\nu_{C=C}$  vibrations of the aromatic rings. A medium intensities bands observed at 1677 cm<sup>-1</sup> and 1617 cm<sup>-1</sup> were attributed, respectively, to the  $\nu_{C=O}$  and  $\nu_{C=N}$  stretching vibrations. The <sup>1</sup>H NMR spectrum recorded in dimethyl sulfoxide (DMSO-d<sub>6</sub>) reveals a singlet signal at 10.7 ppm HO-C=N-OH and a signal at 8.58 ppm due to O=C-N-H showing that in solution, a partial iminolization of the ligand occurred. The signals pointed between 7.39 ppm and 8.06 ppm are attributed to the protons of the aromatic rings. Two singlets pointed at 2.32 ppm and 3.05 ppm are attributed to the proton of the methyl groups and the methylene groups, respectively. The <sup>13</sup>C NMR spectrum of the ligand, presents nine signals. Comparing the <sup>13</sup>C NMR spectrum with that of <sup>13</sup>C{DEPT 135} spectrum, the absence of three signals on the <sup>13</sup>C{DEPT 135} spectrum which were present on the <sup>13</sup>C spectrum is observed. These signals pointed at 175.017 (C=O), 155.637 (C=N) and 148.037 ppm (C<sub>Py-IPSO</sub>) are due to the tetrasubstituted carbon atoms. The signals of the methyl and methylene carbon atoms are pointed at 12.254 ppm and 27.570 ppm, respectively. Four signals, corresponding to the tertiary carbon atoms of pyridine rings, are pointed at 149.024, 137.063, 124.241 and 120.232 ppm, respectively. Comparison of the FTIR spectrum of the H<sub>2</sub>L ligand and those of its complexes reveals a disappearance of the  $\nu_{C=O}$  band and a shift towards low frequencies of the  $\nu_{C=N}$  bands in the spectra of complexes **1-6**. A second  $\nu_{C=N}$  band appears after the iminolization of the ligand in solution. This iminolization is facilitated by the adding LiOH during the reaction to facilitate the deprotonation of the -NH groups (Schema 1). Indeed, we point the  $\nu_{C=N}$  vibrations in the region [1655 cm<sup>-1</sup>–1636 cm<sup>-1</sup>] and [1631 cm<sup>-1</sup>–1593 cm<sup>-1</sup>] for complexes **1-6**. These data confirm the participation of the nitrogen of the imine functions in the coordination to the metal ion. The vibration attributed to  $\nu_{C=N}$  pyridine of the pyridine rings is pointed between 1565 cm<sup>-1</sup> and 1597 cm<sup>-1</sup> on all the spectra of the complexes. For all complexes, the two identical arms of the ligand, acts similarly, and coordinates to two metal ions through the alcoholate oxygen atoms, the azomethine nitrogen atoms and the pyridine nitrogen atoms. The presence of uncoordinated water molecules

is indicated by the  $\nu_{\text{O-H}}$  bands located between  $3435\text{ cm}^{-1}$  and  $3555\text{ cm}^{-1}$  [43].

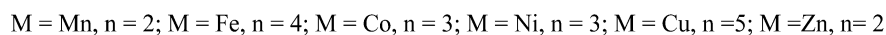
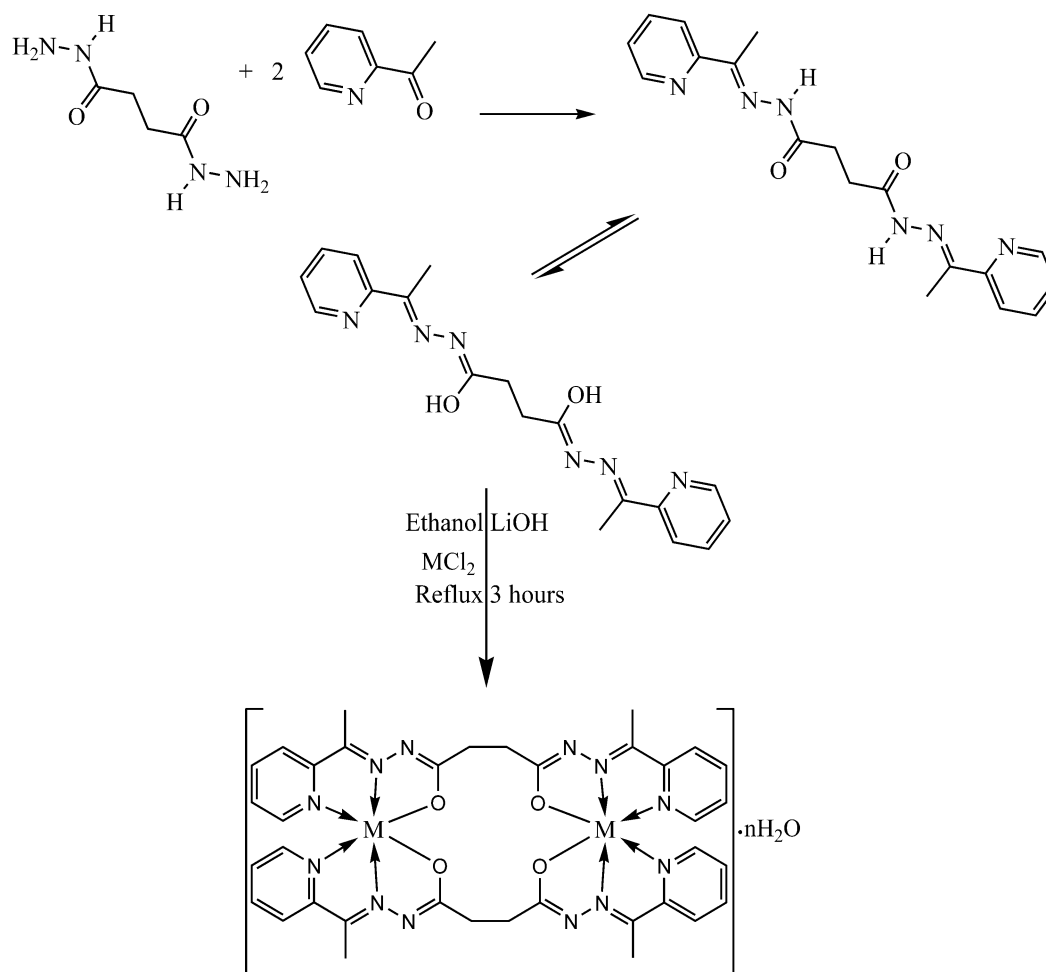
### 3.2 Magnetism

Magnetic study at room temperature has shown that these complexes are binuclear. For the Mn(II) complex (1), the magnetic moment value of  $11.84\ \mu_{\text{B}}$  is consistent with a octahedral dinuclear complex without exchange between the two high-spin centers of  $d^5$  configuration [44]. The Fe(II) complex (2) has a  $\mu_{\text{eff}}$  of  $7.16\ \mu_{\text{B}}$ . This value is close to the spin-only value for two non-coupled high-spin iron(II) [45]. These two ions are located in an octahedral environment. The cobalt complex (3) has two high-spin Co(II) ions in octahedral environment which are magnetically isolated as indicated by the  $\mu_{\text{eff}}$  value of  $7.32\ \mu_{\text{B}}$  [46]. The magnetic

moment value ( $\mu_{\text{eff}} = 5.88\ \mu_{\text{B}}$ ) of complex (4) indicates the presence of two Ni(II) ions in an octahedral environment that are magnetically isolated from each other [47]. Complex (5) is a Cu(II) dinuclear complex without magnetic exchange ( $\mu_{\text{eff}} = 3.92\ \mu_{\text{B}}$ ) in which the two metal ion are located in octahedral geometry [48]. Complex (6) of zinc is diamagnetic as expected for a  $d^{10}$  configuration.

### 3.3 Molar Conductance

All complexes exhibit molar conductivities between 5 and  $60\ \Omega^{-1}\cdot\text{cm}^2\cdot\text{mol}^{-1}$  which indicate that complexes **1-6** have neutral in nature. These values remain almost constant over time, indicating the good stability of the complexes in DMF solutions [49].



**Scheme 1. Synthetic procedure of the ligand  $\text{H}_2\text{L}$  and its metal transition complexes**

**Table 1. Analytical data, room temperature magnetic moments and conductance of complexes 1-6**

Complexes	Yield (%)	Color	% C Calc. (Found)	% H Calc. (Found)	% N Calc. (Found)	$\Lambda$ ( $\Omega^{-1}\cdot\text{cm}^2\cdot\text{mol}^{-1}$ )	$\mu_{\text{eff}}$ ( $\mu_{\text{B}}$ )
H <sub>2</sub> L	89	White	61.35 (61.30)	5.72 (5.70)	23.85 (23.82)	-	-
[(MnL) <sub>2</sub> ] $\cdot$ 2H <sub>2</sub> O (1) C <sub>36</sub> H <sub>40</sub> Mn <sub>2</sub> N <sub>12</sub> O <sub>6</sub>	50	Green	51.07 (51.01)	4.76 (4.71)	19.85 (19.79)	55	11.84
[(FeL) <sub>2</sub> ] $\cdot$ 4H <sub>2</sub> O (2) C <sub>36</sub> H <sub>44</sub> Fe <sub>2</sub> N <sub>12</sub> O <sub>8</sub>	41	Red	48.88 (48.83)	5.01 (4.98)	19.00 (18.93)	27	7.16
[(CoL) <sub>2</sub> ] $\cdot$ 3H <sub>2</sub> O (3) C <sub>36</sub> H <sub>42</sub> Co <sub>2</sub> N <sub>12</sub> O <sub>7</sub>	52	Green	49.55 (49.51)	4.85 (4.83)	19.26 (19.21)	8	7.32
[(NiL) <sub>2</sub> ] $\cdot$ 3H <sub>2</sub> O (4) C <sub>36</sub> H <sub>42</sub> Ni <sub>2</sub> N <sub>12</sub> O <sub>7</sub>	49	Yellow	49.58 (49.55)	4.85 (4.81)	19.27 (19.22)	5	5.88
[(CuL) <sub>2</sub> ] $\cdot$ 5H <sub>2</sub> O (5) C <sub>36</sub> H <sub>46</sub> Cu <sub>2</sub> N <sub>12</sub> O <sub>9</sub>	36	Green	47.11 (47.07)	5.05 (5.01)	18.31 (18.25)	60	3.92
[(ZnL) <sub>2</sub> ] $\cdot$ 2H <sub>2</sub> O (6) C <sub>36</sub> H <sub>40</sub> Zn <sub>2</sub> N <sub>12</sub> O <sub>6</sub>	40	White	49.84 (49.80)	4.65 (4.63)	19.37 (19.33)	5	diam

**Table 2. Main FTIR and UV-visible bands for H<sub>2</sub>L and complexes 1-6**

Compound	$\nu(\text{O-H})$	$\nu(\text{N-H})$	$\nu(\text{N-H})$	$\nu(\text{C=O})$	$\nu(\text{C=N})$	$\nu(\text{C=Npy})$	$\nu(\text{N-N})$	$\lambda(\text{nm})$	
H <sub>2</sub> L		3183	3079	1677	-	1617	1579	1045	210, 265, 296
(1)	3439	-	-	-	1655	1631	1595	1032	213, 263, 296, 345, 446, 537
(2)	3446	-	-	-	1624	1611	1597	1029	208, 261, 295, 342, 395-610
(3)	3531	-	-	-	1652	1620	1595	1044	207, 267, 299, 350, 477, 915
(4)	3459	-	-	-	1650	1600	1595	1041	213, 266, 295, 348, 425, 814
(5)	3455	-	-	-	1658	1613	1596	1033	212, 269, 300, 340, 550-660
(6)	3439	-	-	-	1636	1593	1565	1039	208, 265, 298

### 3.4 Electronic Spectra

The electronic spectra data of H<sub>2</sub>L and the complexes 1-6 are shown in Table 2. The spectrum of the ligand H<sub>2</sub>L exhibits three bands at 210 nm, 265 nm, and 296 nm which were assigned to  $\pi \rightarrow \pi^*$  transitions. These bands are present in the electronic spectra of the complexes 1-6. The complexes 1-5 have an intense absorption band [340 nm – 350 nm] attributable to the metal-to-ligand charge-transfer (MLCT) transition. The Mn(II) complex (1) exhibits two additional bands at 446 nm and 537 nm which are assigned to the  ${}^6A_{1g} \rightarrow {}^4T_{1g}$  and  ${}^6A_{1g} \rightarrow {}^4T_{2g}$  transitions, respectively, corresponding to octahedral geometry for the Mn(II) ion [50]. The Iron complex (2) showed a d-d transition at 395-610 nm attributed to the  ${}^2E_g \rightarrow {}^2T_{2g}$  for d<sup>6</sup> configuration of Fe(II) in an octahedral environment [51]. The electronic spectrum of the cobalt complex (3), displays two bands at 477 nm and 915 nm, which may reasonably be assigned to the  ${}^4T_{1g}(F) \rightarrow {}^4T_{2g}(P)$  and  ${}^4T_{1g}(F) \rightarrow {}^4T_{2g}(F)$  transitions, respectively, suggesting an octahedral geometry around the Co(II) ion [52]. The electronic spectrum of the nickel complex (4), shows two bands located at 425 nm and 814 nm which may be ascribed to the  ${}^3A_{2g}(F) \rightarrow {}^3T_{1g}(P)$  and  ${}^3A_{2g}(F) \rightarrow {}^3T_{1g}(F)$  transitions, respectively indicating an octahedral environment [53,54] around the Ni(II) ion. The UV-visible spectrum of Cu complex (5) showed a broad band in the region 550-660 nm which envelops the three transitions  ${}^2B_{1g} \rightarrow {}^2A_{1g}$ ,  ${}^2B_{1g} \rightarrow {}^2B_{2g}$  and  ${}^2B_{1g} \rightarrow {}^2E_g$ . The broadness of the band is probably due due to the Jahn-Teller effect [55]. These observations suggest that the copper(II) is situated in a distorted octahedral environment [56]. On the basis of elemental analysis, magnetic moment and conductance measurements, UV-Visible spectra and FTIR spectra, each molecule of complexes 1-6 contains two organic ligands acting in hexadentate fashion with two metal ions. Each metal ion is coordinated by two alcoholate oxygen atoms, two nitrogen azomethine atoms and two nitrogen atoms from pyridine ring. Uncoordinated water molecules are present in the structures of the complexes. The suggested structure of the complexes are octahedral in nature as shown in Scheme 1.

### 3.5 Description of the Crystal Structure of the Complex 4

The molecular structure of the nickel(II) complex (4) is given in Fig. 1. Selected bond lengths and

angles are listed in Table 4. The nickel(II) complex crystallizes in the *Pt* space group of the triclinic system. The asymmetric unit of the structure contains two Ni(II) ions and two ligand molecules in its *L*<sup>2-</sup> form. Each Ni(II) ion of the complex (4) is situated in a distorted octahedral geometry, having the N<sub>4</sub>O<sub>2</sub> coordination environment. The coordination of the Ni(II) ion is filled by two O atom of an iminol group, two azomethine nitrogen atoms and two nitrogen atoms from pyridine rings. In each ligand molecule, the flexible alkyl linker chain shows *syn* conformation and the two mono deprotonated tridentate arms of the ligand adopt a *cis* configuration. This behavior of the ligand is observed in similar Schiff base [57]. The structural parameters of the two arms are slightly different. The basal planes of the polyhedron around the Ni1 and Ni2 ions are occupied by O2, N5, N6 and N8 for Ni1 and O4, N2, N10, N11, for Ni2 while the apical positions are occupied by O3 and N7 for Ni1 and O1 and N1 for Ni2. The *cisoid* angles which are in the range [77.64(9)° – 107.71(10)°] and the *transoid* angle 173.28(10)° and 156.59(9)° for Ni1 and 174.37(10)° and 155.72(8)° for Ni2, deviate severely from the ideal values of 90° and 180°, respectively, as expected for a perfect octahedral geometry. The angles subtended by atoms in apical positions [O3—Ni1—N7 = 154.74(9)° and O1—Ni1—N1 = 155.56(8)°] are far from the ideal value of 180°. The octahedral environments around the Ni1 and Ni2 are severely distorted. The sum of the angle subtended by the atoms in the basal planes are 360.08° for Ni1 and 360.16° for Ni2. Each tridentate arm of the ligand forms two five membered rings of type NiNCCN and NiNNCO with the Ni. The chelate ring are quite planar as shown by the small torsion angles (Table 5). The bite angle values resulted are in the range [76.68(9)° – 78.61(10)°]. The mean planes defined by the atoms which form the two six membered ring for each arms of the two ligands are quite perpendicular as shown by the dihedral angle values of 88.58° for Ni1 [Ni1/O3/C26/N9/N8/C24/C23/N7 vs Ni1/O2/C11/N4/N5/C12/C14/N6] and 87.24° for Ni2 [Ni2/O1/C8/N3/N2/C6/C5/N1 vs Ni2/O4/C29/N12/N13/C30/C32/N10]. The apical bond lengths Ni—O [O3—Ni1 = 2.153(2) Å and O1—Ni2 = 2.122(2) Å] are the longest distances around these two nickel atoms. The equatorial bond lengths Ni—N<sub>py</sub> [Ni1—N6 = 2.089(3) Å and Ni2—N10 = 2.082(2) Å] are longer than the equatorial Ni—N<sub>imino</sub> distances which are 1.972(2) Å [Ni1—N5], 2.004(2) Å [Ni1—N8], 1.982(3) Å [Ni2—N2] and 1.978(3) Å [Ni2—N11].

These values are close proximity to the values reported for the complex bis{*N*-[1-(pyridin-2-yl-N)ethylidene]pyridine-4-carbohydrazonato-*k*<sup>2</sup>*N*,*O*}nickel(II)-2,5-dichloroterephthalic acid [58]. The distance Ni—Ni value is 5.991(2) Å. The distances C—O [O1—C8 = 1.252(4) Å, O4—

C29 = 1.257(4) Å, O3—C26 = 1.241(3) Å, O2—C11 = 1.266(3) Å] and C—N [C8—N3 = 1.357(4) Å, C29—N12 = 1.342(3) Å, C26—N9 = 1.364(3) Å, C11—N4 = 1.339(4) Å] are compatible to the values reported with similar ligand [59].

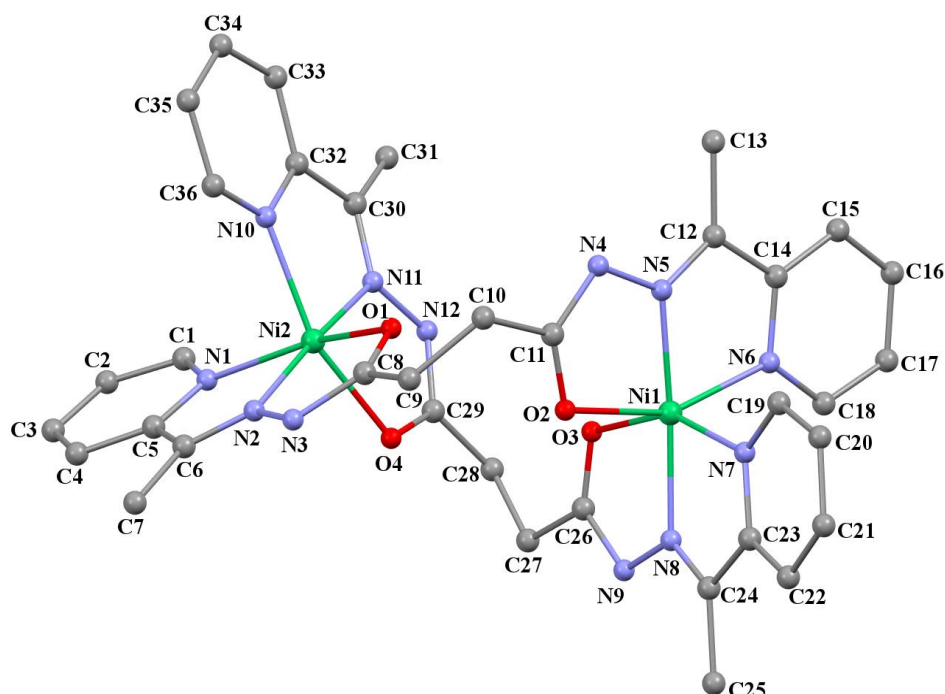


Fig. 1. Crystal structure of the complex (4). Displacement ellipsoids are drawn at the 30% probability level and H atoms are omitted for clarity

Table 3. Crystallographic data and refinement parameters for complex 4

Chemical formula	C <sub>36</sub> H <sub>36</sub> N <sub>12</sub> Ni <sub>2</sub> O <sub>4</sub>
<i>M<sub>r</sub></i>	818.15
Crystal system, space group	Triclinic, <i>P</i> $\bar{1}$
Temperature (K)	173
<i>a</i> , <i>b</i> , <i>c</i> (Å)	10.647 (2), 14.030 (3), 18.077 (4)
$\alpha$ , $\beta$ , $\gamma$ (°)	69.33 (3), 86.34 (3), 70.01 (3)
<i>V</i> (Å <sup>3</sup> )	2369.3 (11)
<i>Z</i>	2
Radiation type	Mo <i>K</i> $\alpha$
$\mu$ (mm <sup>-1</sup> )	0.84
Crystal size (mm)	0.40 × 0.30 × 0.20
No. of measured, independent and observed [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )] reflections	74467, 9777, 8735
<i>R</i> <sub>int</sub>	0.035
<i>R</i> [ <i>F</i> <sup>2</sup> > 2 $\sigma$ ( <i>F</i> <sup>2</sup> )], <i>wR</i> ( <i>F</i> <sup>2</sup> ), <i>S</i>	0.055, 0.174, 1.04
No. of reflections	9777
No. of parameters	491
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\max}$ , $\Delta\rho_{\min}$ (e Å <sup>-3</sup> )	0.62, -0.89

**Table 4. Selected bond lengths (Å) and bond angles (°) of complex 4**

Ni1—N5	1.971 (2)	N5—Ni1—N8	173.28 (10)
Ni1—N8	2.004 (3)	O2—Ni1—N6	156.59 (9)
Ni1—N7	2.069 (3)	N7—Ni1—O3	154.74 (9)
Ni1—O2	2.070 (2)	N5—Ni1—N7	99.35 (10)
Ni1—N6	2.089 (3)	N8—Ni1—N7	78.08 (10)
Ni1—O3	2.153 (2)	N5—Ni1—O2	78.12 (9)
Ni2—N11	1.977 (2)	N11—Ni2—N2	174.37 (10)
Ni2—N2	1.982 (2)	N10—Ni2—O4	155.72 (8)
Ni2—N10	2.082 (3)	N1—Ni2—O1	155.56 (8)
Ni2—N1	2.082 (2)	N11—Ni2—N10	78.20 (9)
Ni2—O4	2.105 (2)	N2—Ni2—N10	106.72 (10)
Ni2—O1	2.122 (2)	N11—Ni2—N1	98.78 (9)

**Table 5. Selected torsion angles (°) in complex 4**

Ni1—O2—C11—N4	1.7(4)
Ni1—N5—C12—C14	0.6(3)
Ni1—N7—C23—C24	6.0(3)
Ni1—O3—C26—N9	-2.8(3)
Ni1—N5—N4—C11	-2.3(3)
Ni1—N6—C14—C12	0.4(3)
Ni1—N8—C24—C23	-0.5(3)
Ni1—N8—N9—C26	-0.3(3)
Ni2—N10—C32—C30	1.4(3)
Ni2—N11—C30—C32	7.9(3)
Ni2—N11—N12—C29	-3.8(3)
Ni2—O4—C29—N12	-0.8(4)
Ni2—N1—C5—C6	-4.5(3)
Ni2—N2—C6—C5	-2.2(4)
Ni2—N2—N3—C8	4.1(3)
Ni2—O1—C8—N3	-2.7(4)

#### 4. CONCLUSION

The new ligand  $N^1$ ,  $N^4$ -bis(1-(pyridin-2-yl)ethylidene)succinohydrazide was synthesized and structurally characterized. The ligand was used for chelation with metal ions with Mn(II), Fe(II), Co(II), Ni(II), Cu(II) and Zn(II) ions. The complexes are characterized by FTIR and UV-visible spectroscopies, room temperature magnetic moments measurements, conductivity measurement and X-ray diffraction for the Ni(II) complexes. The ligand acts as dinegative hexadentate in the all the complexes. In each complex two ligand molecules coordinate two metal ions. Each ligand possesses two arms which coordinates one metal ion in tridentate fashion through one azomethine nitrogen atom, one pyridine nitrogen atom and one iminolate oxygen atom. Octahedral geometries are proposed for the complexes 1-6.

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Author(s) hereby declare that NO generative AI technologies such as Large Language Models (ChatGPT, COPILOT, etc) and text-to-image generators have been used during writing or editing of this manuscript.

#### SUPPLEMENTARY DATA

CCDC-2381435 contains the supplementary crystallographic data for the complex 4. These data can be obtained free of charge via [www.ccdc.cam.ac.uk/conts/retrieving.html](http://www.ccdc.cam.ac.uk/conts/retrieving.html), or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK (Telephone: +44-01223-762910; Fax: +44-1223-336033; or E-mail: [deposit@ccdc.cam.ac.uk](mailto:deposit@ccdc.cam.ac.uk)).

#### IN MEMORIAM

Cheikh Halidou KANE 1963–2021. The death of Dr. KANE has deeply shocked us. Dr. KANE was



a very talented chemist who was deeply involved in research and supervision of doctoral students. His contribution is greatly missed by our team. This article is dedicated to his memory.

## COMPETING INTERESTS

Authors have declared that no competing interests exist.

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